



UNIVERSITI PUTRA MALAYSIA

**PERFORMANCE AND PROPERTIES OF POLYPROPYLENE-
CELLULOSE AND POLYPROPYLENE- OIL PALM EMPTY FRUIT
BUNCH BIOCOMPOSITES**

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AND POLYPROPYLENE- OIL PALM EMPTY FRUIT BUNCH
BIOCOMPOSITES**

By

MOHD KHALID

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirements for the Degree of Master of Science**

January 2007



**DEDICATED
TO
MY FAMILY**

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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January 2007

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Natural fibers such as oil palm empty fruit bunch fibers (EFBF) can be used as environmentally friendly alternatives to conventional reinforcing fibers (e.g., glass) in composites. The interest in natural fiber-reinforced polymer composites is growing rapidly due to its high performance in terms of mechanical properties, significant processing advantages, excellent chemical resistance, low cost and low density. These advantages place natural fiber composites among the high performance composites having economic and environmental advantages. On the other hand, lack of good interfacial adhesion and poor resistance to moisture absorption make the use of natural fiber-reinforced composites less attractive. In order to improve their interfacial properties, these EFB fibers were subjected to chemical treatments, namely, chlorination, mercerization and acetylation. Preparation of cellulose by selective removal of non-cellulosic compounds constitutes the main objective of the chemical treatments of EFBF to improve the performance of fiber-reinforced composites. The objective of this study was to determine the effects of cellulose on the performance of the cellulose-reinforced biocomposites and comparing its property with the EFBF-

reinforced biocomposites. Biocomposites were prepared by blending polypropylene-cellulose and polypropylene-EFBB at different weight ratios using a twin screw brabender. Further, effects of two different coupling agents namely MAPP and TMPTA on the properties of PP-cellulose and PP-EFBB biocomposite were also studied. These coupling agent were incorporated in order to enhance the fiber matrix adhesion. Mechanical and physical properties of both the biocomposites were evaluated. Compared to PP-EFBB biocomposites, PP-cellulose biocomposites showed better fiber-matrix interaction as observed from the good dispersion of fibers in the matrix system. The tensile fracture and impact fracture surfaces of the composites were characterized by scanning electron microscopy confirms the cellulose and PP interface had improved interfacial bonding. Incorporation of MAPP as coupling agent does not show significant improvement in case of PP-cellulose biocomposite. However, it showed good results for PP-EFBB biocomposite. On the other hand TMPTA coupled PP-cellulose biocomposite offered superior physical and mechanical properties. The strong intermolecular cellulose-matrix bonding indicates a decrease in the high rate of water absorption in PP-cellulose biocomposites. The dynamic mechanical analysis (DMA) and Thermogravimetry analysis (TGA) technique were also used to measure the viscoelastic properties and melting point of both the biocomposite. The scanning electron microscopy photographs of fiber surface characteristics and fracture surfaces of composites clearly indicated the extent of fiber-matrix interface adhesion.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains.

**PRESTAI DAN SIFAT BIOKOMPOSIT SELULOSA-POLIPROPELENA DAN
SERABUT TANDAN KOSONG MINYAK KELAPA SAWIT-
POLIPROPELENA**

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Serabut asli seperti serabut tandan kosong minyak kelapa sawit (EFBF) boleh digunakan sebagai alternatif komposit yang mesra alam bagi menggantikan serabut penguatan konvensional (contoh: kaca). Minat terhadap komposit polimer penguat-serabut asli berkembang dengan pesatnya kerana prestasi yang tinggi dari segi sifat mekanik, faedah pemprosesan yang penting, ketahanan kimia yang cemerlang, harga rendah dan ketumpatan rendah. Faedah-faedah ini meletakkan serabut asli antara komposit yang tinggi kecekapan yang mempunyai faedah dari segi alam sekitar dan ekonomik. Dalam pada itu, kekurangan dari segi lekatan antara muka dan rintangan terhadap serapan kelembapan menyebabkan penggunaan komposit penguat-serabut asli kurang mendapat tarikan. Untuk memperbaiki sifat-sifat antara muka mereka, serabut tandan kosong (EFBF) bergantung kepada rawatan kimia iaitu pengklorinan, penggilapan dan pengasetilan. Penyediaan selulosa dengan cara pembuangan sebatian bukan selulosa yang terpilih menjadikan objektif utama rawatan kimia EFBF untuk memperbaiki kecekapan komposit penguat-serabut. Objektif kajian ini adalah untuk menentukan kesan-kesan selulosa terhadap keupayaan biokomposit penguat-selulosa

dan membandingkan sifat tersebut dengan biokomposit penguat-EFBB. Biokomposit disediakan dengan mengadunkan selulosa-polipropelena (PP) dan EFBB-polipropelena pada nisbah berat yang berlainan menggunakan *brabender* skru berkembar. Tambahan lagi, kesan daripada dua agen pengganding yang berbeza yang dinamakan MAPP dan TMPTA terhadap sifat-sifat biokomposit selulosa-PP dan EFBB-PP juga dikaji. Agen pengganding ini digabungkan untuk menambah lekatan matrik serabut. Sifat-sifat mekanik dan fizikal untuk kedua-dua biokomposit dinilai. Berbanding dengan biokomposit EFBB-PP, biokomposit selulosa-PP menunjukkan saling tindak matrik-serabut yang lebih baik setelah diperhatikan dari segi serakan serabut yang baik di dalam sistem matrik. Kepatahan tegangan dan hentaman terhadap permukaan komposit digambarkan dengan mikroskopi elektron pengimbasan (SEM) yang mengesahkan ikatan antara muka di antara PP dan selulosa telah diperbaiki. Penggabungan MAPP sebagai agen pengganding tidak menunjukkan sebarang pembaikan yang bernilai bagi kes biokomposit selulosa-PP. Walaubagaimanapun, ia menunjukkan keputusan yang bagus pada biokomposit EFBB-PP. Dalam pada itu, biokomposit selulosa-PP terganding TMPTA menawarkan sifat-sifat mekanik dan fizikal yang lebih baik. Ikatan matrik-selulosa antara molekul yang kuat menunjukkan pengurangan di dalam kadar serapan air yang tinggi pada biokomposit selulosa-PP. Teknik analisis mekanik dinamik (DMA) dan analisis termogravimetri (TGA) juga digunakan untuk mengukur sifat-sifat likat anjal dan takat lebur untuk kedua-dua biokomposit. Keseluruhannya, fotograf SEM untuk ciri-ciri permukaan serabut dan permukaan kepatahan oleh komposit dengan jelasnya menunjukkan had lekatan antara muka matrik-serabut.

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Lastly with unquantifiable affection and reference I wish to express my sincere feeling to my parents and my wife in the form of words which are rather restrictive in expression of quantum.

I certify that the Examination Committee met on 19/01/2007 to conduct the final examination of Mohd. Khalid on his Master of Science in Environmental Engineering thesis entitled “Performance and Properties of Polypropylene-Cellulose and Polypropylene- Oil Palm Empty Fruit Bunch Biocomposites ” in accordance with Universiti Pertanian Malaysia (Higher degree) Act 1980 and Universiti Pertanian Malaysia (Higher degree) Regulation 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

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TABLE OF CONTENTS

		Pages
	DEDICATION	ii
	ABSTRACT	iii
	ABSTRAK	v
	ACKNOWLEDGEMENTS	vii
	APPROVAL	viii
	DECLARATION	x
	LIST OF TABLES	xiv
	LIST OF FIGURES	xv
	LIST OF NOTATIONS AND ABBRIVATIONS	xix
	 CHAPTER	
1	INTRODUCTION	
	1.1 Background	1.1
	1.2 Characteristics of Thermoplastic Polymers	1.4
	1.3 Malaysian Scenario	1.4
	1.4 Application of Biocomposites	1.5
	1.5 Problem Statement	1.6
	1.6 Hypothesis	1.6
	1.7 Scope of Study	1.7
	1.8 Objectives	1.7
	1.9 Structure of Thesis	1.8
 2	 LITERATURE REVIEW	
	2.1 Natural Fibres for Reinforcement	2.1
	2.2 Properties of Natural Fibers	2.2
	2.2.1 Physical Properties of Natural Fibers	2.2
	2.2.2 Chemical Composition of Natural Fibers	2.5
	2.2.3 Mechanical Properties of Thermoplastic-Natural Fibers Biocomposites	2.10
	2.2.4 Water Absorption Characteristics Thermoplastic-Natural Fibres biocomposites	2.11
	2.3 Technicalities of Cellulose Fibers-Thermoplastic Biocomposites.	2.12
	2.3.1 Fiber Dispersion	2.14
	2.3.2 Fiber-Matrix Adhesion and Interaction	2.15
	2.3.3 Fiber Aspect Ratio	2.18
	2.3.4 Fiber Orientation	2.21
	2.3.5 Fiber Volume Fraction	2.23

2.4	Surface Chemical Modifications of Natural Fibers	2.25
2.4.1	Effect of Alkali Treatment (Mercerization) on Natural Fiber	2.25
2.4.2	Effect of Crosslinking Agents on Biocomposites	2.29
2.4.3	Other Treatment Method	2.30
2.5	Effects of Fiber Surface Modifications on Lignocellulosic Fibers	2.31
2.5.1	Stress-Strain Behaviour	2.31
2.5.2	Tensile Properties of Fibers	2.32
2.6	Processing Considerations and Techniques	2.33
2.7	Effects of Fiber Surface Modifications on Biocomposite Properties	2.37
2.7.1	Mechanical Properties of Biocomposites	2.37
2.7.2	Thermal Properties of Biocomposites	2.40
2.7.3	Dynamic Mechanical Analysis (DMA)	2.40
2.7.4	Macro-Mechanical Properties of Biocomposites	2.42
2.8	Summary	2.42
3	METHODOLOGY	
3.1	Materials	3.1
3.1.1	Empty Fruit Bunch Fiber (EFBF)	3.1
3.1.2	Thermoplastic	3.2
3.1.3	Chemical and Coupling Agents	3.2
3.2	Cellulose Preparation	3.3
3.3	Biocomposite Preparation	3.4
3.4	Biocomposite Sample Preparation	3.6
3.5	Mechanical Properties of the Biocomposite	3.7
3.5.1	Tensile Strength of Biocomposites	3.7
3.5.2	Flexural Strength of Biocomposites	3.7
3.5.3	Impact Strength of Biocomposites	3.8
3.5.4	Dynamic Mechanical Analysis (DMA)	3.10
3.6	Physical Properties	3.11
3.6.1	Hardness	3.11
3.6.2	Water Absorption	3.12
3.6.3	Melt Flow Index (MFI)	3.13
3.7	Thermal Analysis	3.14
3.7.1	Thermogravimetric Analysis (TGA)	3.14
3.8	Interfacial Morphology Analysis (SEM)	3.15
4	RESULTS AND DISCUSSIONS	
4.1	Mechanical Properties	4.1
4.1.1	Tensile Strength of Biocomposites	4.2
4.1.2	Flexural Modulus of Biocomposites	4.5
4.1.3	Impact Strength of Biocomposites	4.8
4.1.4	Rockwell Hardness of Biocomposites	4.11
4.1.5	Correlation between Fiber Structure and Mechanical Properties	4.13
4.2	Physical Properties	4.14

4.2.1	Water Absorption of Biocomposites	4.15
4.3	Flow Property	4.18
4.3.1	Melt Flow Index (MFI)	4.18
4.4	Thermal Analysis	4.21
4.4.1	Thermogravimetry Analysis (TGA)	4.21
4.5	Dynamic Mechanical Analysis (DMA)	4.27
4.6	SEM Morphological Study	4.33
5	CONCLUSIONS AND RECOMMENDATION	
5.1	Conclusions	5.1
5.2	Recommendations	5.2
	REFERENCES	R.1
	BIODATA OF THE AUTHOR	B.1
	LIST OF PUBLICATIONS	L.1

LIST OF TABLES

Table	Page
2.1 Morphological Properties of Oil Palm Fiber in Comparison with Hardwood and Softwood	2.3
2.2 Characteristic Values for the Density, Diameter and Mechanical Properties of Natural and Synthetic Fibers	2.4
2.3 Chemical Composition, Moisture Content and Microfibrillar Angle of Natural Fibers	2.9
2.4 Summary of Previous Studies on the Natural Fiber Composites, Different Chemical Treatments and Coupling Agents	2.43
3.1 Composition of PP-Cellulose and PP-EFBF Biocomposite	3.5
3.2 Composition of PP-Cellulose and PP-EFBF Biocomposite with Coupling Agent	3.6
4.1 Summary of DTG _{max} Degradation Temperature of PP-Cellulose and PP-EFBF Biocomposite	4.26
4.2 Summary of Tan δ_{max} Peak Temperature of Biocomposites	4.33

LIST OF FIGURES

Figure		Page
1.1	Classification of Natural Fibers Which Can be Used as Fillers and Reinforcers in Polymer	1.2
2.1	Positioning of the Cellulose Fibrils in Wood and Cotton Fibers	2.5
2.2	Schematic Diagram of Cellulose Molecule	2.6
2.3	Variation in the Strength And Stiffness of Jute Fibers with Lignin Content	2.8
2.4	Variation in the Strength And Stiffness of Jute Fibers with Lignin Content	2.31
2.5	Fiber Tensile Stress and Shear Stress Variation Along the Length	2.19
2.6	Stress–Position Profiles with Fiber Length	2.20
2.7	Deformation Pattern in the Matrix Surrounding a Fiber	2.20
2.8	Schematic Representations of the Changes in Fiber Orientation Occurring During Flow	2.22
2.9	Illustration of Four Stages of Deformation of Fibers, Matrix and Composite	2.23
2.10	Typical Relationships Between Tensile Strength and Fiber Volume Fraction for Short Fiber-Reinforced Composites	2.24
2.11	Detailed Chemical Structure of a Microfibril	2.27
2.12	Detailed structural Changes of Microfibrils During Mercerization	2.28
3.1	Raw Material Empty Fruit Bunch (EFB) and Processed Empty Fruit Bunch Fibers (EFBF) of Oil Palm	3.1
3.2	Polypropylene Pellets	3.2
3.3	Production of Cellulose from EFBF by Two Stage Chemical Treatment.	3.4

3.4	Production of PP-Cellulose and PP-EFBF Biocomposites	3.5
3.5	Preparation of PP-Cellulose and PP-EFBF Biocomposites Samples	3.7
3.6	Instron Universal Testing Machine for Tensile and Flexural Testing	3.8
3.7	Ceast-Impact Pendulum Apparatus for Impact Testing	3.9
3.8	Perkin-Elmer DMA apparatus for dynamic mechanical analysis	3.11
3.9	Rockwell Hardness Apparatus for Hardness Measurement	3.12
3.10	Water Absorption Test for Biocomposites	3.13
3.11	Melt Flow Apparatus for MFI Measurements	3.14
3.12	Perkin-Elmer TGA apparatus for thermogravimetric analyses	3.15
4.1	Effect of Filler Loading on the Tensile Strength of PP-Biocomposites	4.2
4.2	Effect of APP o Tensile Strength o PP-Biocomposites a 30 Wt % Filler Loading	4.4
4.3	Effect of TMPTA on Tensile Strength of PP-Biocomposites at 30 Wt % Filler Loading	4.5
4.4	Effect of Filler Loading on Flexural Modulus of PP-Biocomposites	4.6
4.5	Effect of MAPP on Flexural Modulus of PP-Biocomposites at 30 Wt % Filler Loading	4.7
4.6	Effect of TMPTA on Flexural Modulus of PP-Biocomposites At 30 Wt % Filler Loading	4.8
4.7	Effect of Filler Loading on Impact Strength of PP-Biocomposites	4.9
4.8	Effect of MAPP on Impact Strength of PP-Biocomposites at 30 wt % Filler Loading	4.10
4.9	Effect of TMPTA on Impact Strength of PP-Biocomposites at 30 wt % Filler Loading	4.10
4.10	Effect of Filler loading on Hardness of PP-Biocomposites	4.11
4.11	Effect of MAPP on the Hardness of PP-Biocomposites at 30 wt % Filler Loading	4.12
4.12	Effect of TMPTA on the Hardness of PP-Biocomposites at 30 wt % Filler Loading	4.13
4.13	Effect of Filler Loading on Water Absorption of PP-Biocomposites	4.16

4.14	Effect of MAPP on Water Absorption of PP-Biocomposites at 30 Wt % Filler Loading	4.17
4.15	Effect of TMPTA on Water Absorption of PP-Biocomposites at 30 Wt % Filler Loading	4.18
4.16	Effect of Filler Loading on Melt Flow Index of PP-Biocomposites	4.19
4.17	Effect of MAPP on Melt Flow Index of PP Biocomposites at 30 Wt % Filler Loading	4.20
4.18	Effect of TMPTA on Melt Flow Index of PP Biocomposites at 30 Wt % Filler Loading	4.21
4.19	Thermogravimetry Analysis of PP Cellulose And EFBF	4.22
4.20	Derivative Thermogravimetry Analysis of PP Cellulose and EFBF	4.23
4.21	Thermogravimetry Curves of PP-Cellulose and PP-EFBF Biocomposites	4.24
4.22	Derivative Thermogravimetry Curves of PP-Cellulose and PP-EFBF Biocomposites	4.25
4.23	Hypothetical Model of the Thermal Degradation of Cellulose	4.25
4.24	Effect of Cellulose Loading on Storage Modulus of PP Biocomposites	4.28
4.25	Effect of Filler Loading on Loss Modulus of PP Biocomposites	4.29
4.26	Effect of Filler Loading on Tan Delta of PP Biocomposites	4.30
4.27	Effect of MAPP on Tan Delta of PP-Cellulose Biocomposites at 30 Wt % Cellulose Loading	4.32
4.28	Effect of TMPTA on Tan Delta of PP-Cellulose Biocomposites at 30 Wt % Cellulose Loading	4.33
4.29	SEM Micrograph Showing Tensile Fracture Surface of 30 Wt % PP-EFBF Biocomposite	4.34
4.30	SEM Micrograph Showing Tensile Fracture Surface of 50 Wt % PP-EFBF Biocomposite	4.35
4.31	SEM Micrograph Showing Tensile Fracture Surface of 30 Wt % PP-Cellulose Biocomposite	4.36
4.32	SEM Micrograph Showing Tensile Fracture Surface of 50 Wt % PP-Cellulose Biocomposite	4.37

4.33	SEM Micrograph Showing Impact Fracture Surface of 30 Wt % PP-EFBF Biocomposite	4.38
4.34	SEM Micrograph Showing Impact Fracture Surface of 30 Wt % PP-Cellulose Biocomposite	4.38
4.35	SEM Micrograph Showing Effect of 2 Wt % MAPP on PP-EFBF Biocomposite at 30 Wt % EFBF Loading	4.40
4.36	SEM Micrograph Showing Effect of 2 Wt % MAPP on PP-Cellulose Biocomposite at 30 Wt % Cellulose Loading	4.40
4.37	SEM Micrograph Showing Effect of 2 Wt % TMPTA on PP-EFBF Biocomposite at 30 Wt % EFBF Loading	4.41
4.38	SEM Micrograph Showing Effect of 2 % TMPTA on PP-Cellulose Biocomposite at 30 Wt % Cellulose Loading	4.42
4.39	SEM Micrograph Showing Effect of 7 Wt % MAPP on PP-EFBF Biocomposite at 30 Wt % EFBF Loading	4.43
4.40	SEM Micrograph Showing Effect of 7 Wt % MAPP on PP-Cellulose Biocomposite at 30 Wt % Cellulose Loading	4.44
4.41	SEM Micrograph Showing Effect Of 7 Wt % TMPTA on PP-EFBF Biocomposite at 30 Wt % EFBF Loading	4.44
4.42	SEM Micrograph Showing Effect of 7 Wt % TMPTA on PP-Cellulose Biocomposite at 30 Wt % Cellulose Loading	4.45

ABBREVIATIONS

ΔH_f	Heat of Fusion
CrR	Crystallinity Ratio
d	Diameter
E^*	Storage Modulus
E'	Loss Modulus (E'')
fr	Hermans factor
l	Fiber Length
l_c	Critical Length
$\tan \delta$	Mechanical Damping
T_c	Crystallization Temperature
T_g	Glass Transition Temperature
V_c	Critical Fiber Volume Fraction
X_c	Crystallinity
α	Melting Temperature
β	Glass-Rubbery Transition
σ	Tensile Strength
σ_f^*	Fiber Tensile Strength
σ_{fu}	Fiber Ultimate Strength in Tension
τ_y	Interfacial Shear Stress
ASTM	American Society for Testing and Materials

BC	Bacterial Cellulose
CPO	Crude Palm Oil
DMA	Dynamic Mechanical Analysis
DP	Degree of Polymerization
DTG	Degradation Temperature
EFBF	Empty Fruit Bunch Fiber
EHMA	Ethyl α -HydroxyMethylAcrylate
FFB	Fresh Fruit Bunch
HDPE	High-density polyethylene
ION	Ionomer-Modified Polyethylene
ISS	Interfacial Shear Strength
LDPE	Low-density polyethylene
LWMPP	Low Molecular Weight Polypropylenes
MAPP	Maleic Anhydride Modified Polypropylene
MFI	Melt Flow Index
MMA	Methyl Methacrylate
MPOB	Malaysian Palm Oil Board
OPF	Oil Palm Fronds
POME	Palm Oil Mill Effluent
PP	Polypropylene
RGP	Refiner Ground Pulp
RH	Relative Humidity
SEM	Scanning Electron Microscope
TG	Thermogravimetric
TGA	Thermogravimetic Analysis

TMPTA	Trimethylolpropane Triacrylate
WPC	Wood-Polymer Composites

CHAPTER 1

INTRODUCTION

1.1 Background

Over the last decade, a rapid growth occurred in the consumption of the plastic products in various fields. However, due to diminution and escalating price of petroleum based products, the shortage of landfill space, concern over emission during incineration and entrapment by ingestion of packaging plastic by fish, fowl and animals has spurred efforts to explore and develop better alternatives that are compatible with the environment and independent of fossil fuel. More down-to-earth, however, is the fact that our society has become very energy conscious. This also has increased the demand for lightweight yet strong and stiff structures in all walks of life. Composites, especially polymers reinforced with natural fibers have received growing interest, both from the academic world and from various industries. There is a wide variety of different natural fibers which can be applied as reinforcers or fillers. An illustration with a classification of the various fibers is presented in Figure 1.1. All these natural fibers consist of long cells with relatively thick cell walls which make them stiff and strong. The chemical composition as well as the structure of plant fibers is fairly complicated. Plant fibers are composite material designed by nature. The fibers are basically a rigid, crystalline cellulose microfibril reinforced amorphous lignin and/or hemicelluloses matrix. Therefore, cellulose makes the principal component of plant fiber where it provides the main structural feature. These fibers sometimes are referred as lignocellulosic fibers due to the presence of lignin and hemicellulose. The most important of the natural fibers used in composite materials

are flax, hemp, jute, kenaf sisal and empty fruit bunch fibers, due to their good properties and availability.

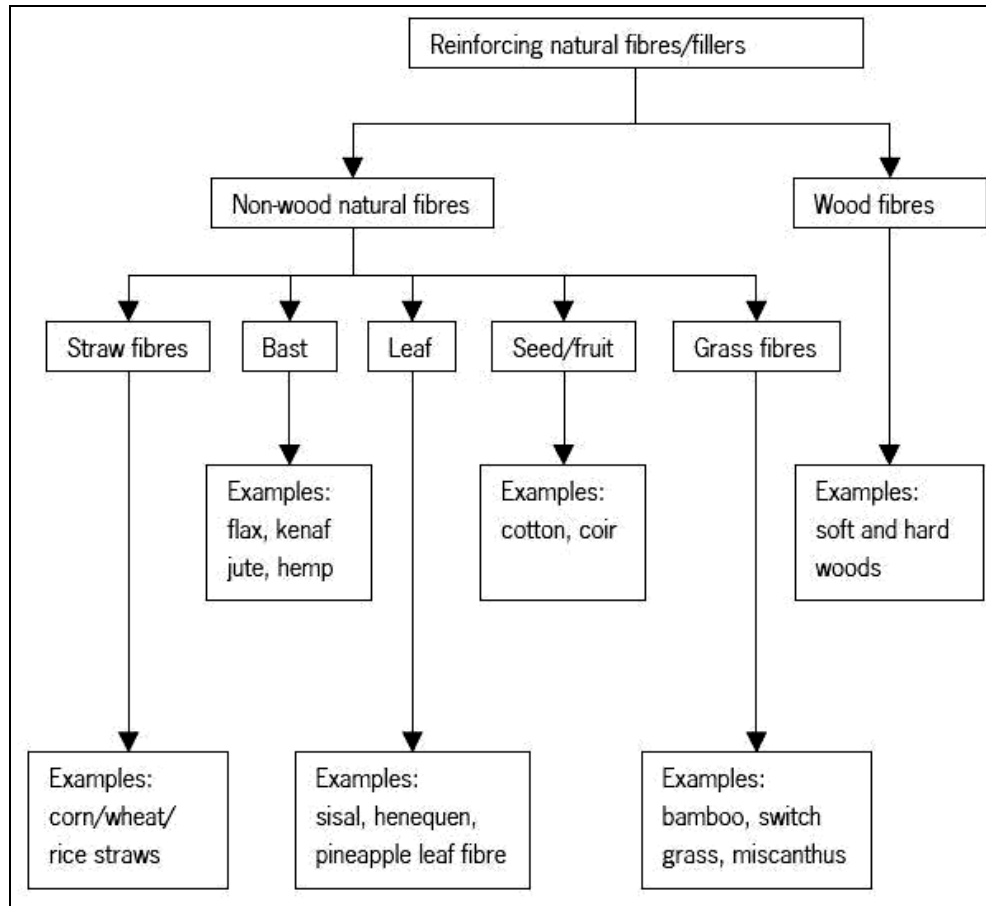


Figure 1.1 Classification of natural fibers which can be used as fillers and reinforcers in Polymer (Mohanty *et al.*, 2002).

Generally, four main reasons are mentioned which make the application of natural fibers attractive: (1) their specific properties, (2) their price, (3) their health advantages and (4) their recyclability. Natural fibers based on cellulose have a relatively low density, and are relatively stiff and strong. Therefore their specific properties are rather high, and actually comparable to those of conventional reinforcing fibers. Though, the environmental driving force has never been as important as it is in today's scenario. Natural fiber reinforced composites are

originally aimed at the replacement of glass fiber and other inorganic fiber reinforced composites. A specific advantage of fiber composites over glass fiber composites, however, is the fact that they can be burned (thermal recycling) without leaving large amounts of slag. On the whole, the use of natural fibers has a definite 'green image'. For the automotive industry, for instance, this has been a serious driving force for the development of natural fiber reinforced materials and it has also induced companies like DaimlerChrysler AG, Mercedes and Ford to try and develop high performance materials on the basis of renewable resources (Mapelstone, 1999; Broge, 2000).

Considering all the above advantages natural fiber-reinforced thermoplastic composites eventually form a new class of materials. This seems to have a good potential in the future as a substitute for wood and petroleum based material in numerous applications. But, in reality lack of good interfacial adhesion and poor resistance to moisture absorption makes the use of natural fiber-reinforced composites slightly tedious. To overcome these problems various fiber surface treatments like mercerization, isocyanate treatment, acrylation, latex coating, permanganate treatment, acetylation, silane treatment and peroxide treatment have been set up which may result in improving composite properties. Reinforcing fibers are normally given surface treatments to improve their compatibility with the polymer matrix as interfaces play an important role in the physical and mechanical properties of composites. Research on a cost effective modification of natural fibers is necessary since the main attraction for today's market of biocomposites is the competitive cost of natural fiber.